Practical Application of the EVT Concept

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Abstract

In 1999, the Canadian Roofing Contractors' Association (CRCA) initiated a study to compare the quality and performance properties of bulk asphalt from various suppliers/manufacturers across Canada. The study was prompted by concerns raised by contractor members about the quality of bulk asphalt and, in particular, fallback (softening point drop back) after heating the asphalt during built-up roofing (BUR) applications. The study consisted of a quantitative analysis of changes to the physical properties of asphalt samples, including equiviscous temperature (EVT), after heating and assessment of the quality of the information provided to roofing contractors by asphalt suppliers/manufacturers regarding their recommended EVTs. The issue of the quality of EVT information is discussed in ***Appendix A*** of this paper. The concept of EVT is widely accepted as a quality control instrument in the application of BUR systems. The study was designed to assess the effectiveness of the concept as currently applied in the field.

Authors Biographies

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Background

In the construction of BUR systems, it is necessary to heat the asphalt to temperatures at which it becomes a liquid of sufficiently low viscosity so it can be poured, mopped or dispensed by mechanical spreaders. Prior to heating asphalt to the required temperatures, the material typically is delivered to a job site either as a solid in the form of kegs or blocks or as a viscous liquid in tankers where it then is transferred to kettles.

Since its introduction in late 1977, the concept of EVT has been widely accepted throughout the North American roofing industry as a means of providing an application temperature window and in-the-field quality control.[1] Before the introduction of EVT, manufacturers' recommended application temperatures were based on empirically determined temperature limits intended to obtain the ad hoc standard interply weight of mopping asphalt of 1.2 kg/m^2 (25 pounds/100 square feet). Maximum heating temperatures addressed safety considerations (e.g. flash point) and the prevention of fallback of the particular asphalt's softening point caused by overheating. These upper limits guided most BUR applications, and the asphalt typically was heated to the highest allowable temperature. There were no universally accepted maximum heating temperatures for each asphalt type. Recommended limits ranged from a low of 220°C (428°F) to a high of 260°C (500°F). [2,3]

Experience showed that strict adherence to these arbitrary maximums resulted in many BUR membranes being constructed with asphalt being applied at temperatures too low to obtain adequate adhesion between the asphalt and reinforcing plies. Several potential problems were identified with asphalt applied at too low a temperature. Too much applied between the reinforcing layers can increase slippage potential, inadequate adhesion, void formation between plies and subsequent blister formation.[4] Too little can yield skipped areas and poor adhesion. These potential problems can be exacerbated in cold weather due to the rapid chilling of the asphalt.

In their paper published in 1976, Rossiter and Mathey demonstrated that the application of asphalt should be based on viscosity, the property of the asphalt which governs its flow.[5] When asphalt is used to laminate reinforcing felts, it should be applied at temperatures at which it has attained a viscosity that will ensure that the quantity of interply asphalt will be at the recommended quantity of 1 kg/m^2 to 2 kg/m^2 (20 pounds/square foot to 40 pounds/square foot) regardless of the type of roofing asphalt used, provided that the proper application techniques are followed. The ideal apparent viscosity of roofing asphalts for mop applications, as determined by ASTM D-4402, "Test Method for Viscosity Determinations of Unfilled Asphalts Using the Brookfield Thermoset Apparatus," is 125cP (0.125 Pa·s), and 75cP (0.075 Pa·s) for mechanical spreader applications.[6] Industry research and practice indicate that asphalts applied within the EVT application range will result in a homogeneous roof membrane assembly exhibiting the desired adhesion of the bitumen to the reinforcing ply sheets.

When a BUR system is being constructed, it is the responsibility of the kettle operator to maintain the asphalt at the proper temperature so as to achieve the desired EVT at the point of application. Factors that may affect heating temperatures, other than the asphalt type, are weather conditions, distance of the kettle from application location, and type and condition of the heating equipment. Maintaining asphalt temperatures within the EVT range may be difficult when the roof is applied in cold weather. Rapid cooling of the hot asphalt may result in poor adhesion, voids in the asphalt layers or excessive and nonuniform applied thickness between the layers of reinforcing material. Rossiter et al have demonstrated that cooling times vary widely with the type of substrate to which the asphalt is applied and environmental conditions. Rossiter states:

"Under certain environmental conditions, hot bitumen applied to some substrates cools extremely rapidly. In these cases, sufficient time for proper applications may not be available." [7]

Dupuis et al reported that during asphalt application in the field, peak contact temperatures were usually 28 to 42°C (50 to 75°F) lower than the temperature in the asphalt kettle.[8] During field investigations, they recorded asphalt cooling times at ambient temperatures ranging from -7°C to 4°C (20-39°F) and with wind speeds of 1.4 m/s to 6.3 m/s (3 mph to 14 mph). Under these conditions when asphalt was applied to the insulation, the temperature decreased by approximately 28°C (50°F) in 2 to 7 seconds. Their findings indicate that raising the peak contact temperature, and consequently the kettle temperature during cold weather, may be the only effective means to increase the time the asphalt remains sufficiently hot to adhere the felts properly. The dilemma for the kettle operator, especially during cold weather applications, always has been how to obtain and maintain the asphalt at temperatures within the EVT range at the point of application but not so high as to cause material degradation.

Asphalt Specifications

The temperature at which the bitumen attains the optimal viscosity is determined by the chemical composition of the material. According to the Canadian Standard for Asphalt, CSA 123.4-98, each supplier/manufacturer is to provide information regarding the recommended EVT on the asphalt packaging, or in the case of bulk asphalt, on the bills of lading.[9] Manufacturers are required to report only the EVT for the viscosity of 125cP associated with hand mopping. The standard defines EVT as the temperature at which the viscosity of the asphalt is 125 mm^2/s (125cP); the recommended asphalt temperature ± 14°C (± 25°F) at the time of application to the substrate. EVT is to be measured using ASTM Standard D4402. If an alternate test method is used, it must be reported.

Analysis and Testing

The study was carried out in two phases. In Phase I, asphalt samples obtained from contractors were heated under laboratory conditions at a temperature of 260±10°C (500±18°F) for four hours. In Phase II, samples were heated under similar conditions at a temperature of 232±10°C (450±18°F) for four hours. The first temperature (260±10°C [500±18°F]) and heating time were selected to simulate the most severe heating conditions that would be encountered during cold weather roofing and application by both hand mopping and mechanical spreader. The second temperature (232±10°C (450±18°F]) and heating time were selected to simulate roof applications under ideal summer conditions.

Janicki claimed that the softening point of asphalt increased when the kettle was kept open and decreased when the kettle was kept closed.[10] The author explained that when the kettle was kept open, the light oils in the asphalts were evaporated. This led to an increase in softening point and the asphalt became harder and more brittle. When the kettle was kept closed, the light oils in the asphalt were boiled off, condensed on the inside of the kettle and remixed with the bulk. This reflux action reduced the softening point of the asphalt. In this study the samples were heated under open and closed conditions to ascertain any difference on their softening point and EVT after exposure.

Phase I--Sampling and Testing

In May and June 1999, CRCA received samples of Canadian Type 2 roofing asphalt from roofing contractors located in western and central Canada. Canadian Type 2 asphalt was selected for analysis because it is the most common type used with tanker operations. The samples were obtained directly from tankers filling up at five different supply locations and transferred into 1-gallon metal cans. After having cooled to solid form, the samples, weighing about 5 kg (10 pounds) each, were shipped to CRCA. CRCA delivered the samples to National Research Council's Institute for Research in Construction for analysis.

Each asphalt sample was divided into three portions Control, Open and Closed. The Control samples were maintained as "as received" condition to provide baseline data for comparison. They received minimal heating just enough to allow proper pouring for specimen preparation. The other two portions were heated in half-filled 1-litre tin cans with and without lids in a convection oven at 260±10°C (500±18°F) for four hours to simulate the open and closed kettle operations in the field. They are referred to Open (without lid) and Closed (with lid) samples. A thermocouple was placed in the asphalt in each can during the heating process for temperature monitoring. Test specimens were prepared from the hot asphalt immediately after the four-hour heating. The heating conditions are summarized as follows:

Sample	**Heating Condition**
Control	Heated in 1-L tin cans at 140°C (284°F) for 3-4h in a convection oven until liquid enough for pouring
Open	Heated in 1-L cans (without lids) at 260±10°C (500±18°F) for 4h in a convection oven
Close	Heated in 1-L cans (with lids) at 260±10°C (500±18°F) for 4h in a convection oven

In Phase I, the softening points of the samples were determined according to the ASTM D36, Standard Test Method for Softening Point of Bitumen (Ring and Ball Apparatus).[11]

Rheological Properties--Viscosity

The viscosity of the asphalt samples was determined in accordance with ASTM D4402-87, Test Method for Viscosity Determinations of Unfilled Asphalts Using the Brookfield

Thermoset Apparatus, using a Bohlin Visco 88 viscometer equipped with a #2 cup and spindle.[12] The viscometer was calibrated with Brookfield viscosity standards of 50, 100 and 500cP. The temperature of the sample was controlled using either an oil bath or a sand bath during the measurement. By varying the shear rate at a particular temperature, the corresponding shear stresses were recorded. The viscosity of the asphalt at that temperature then was determined by linear regression through the shear stress and shear rate data. By adjusting the temperature of the heating bath, the viscosity, as a function of temperature, was determined for each asphalt sample.

Results--Softening Point

The softening points of the five asphalt samples with three different heating histories are summarized in Table 1. The softening points of the five asphalt samples varied between 75°C and 86°C (167°F to187°F). The softening points of Samples B, C and E were within the CSA specification for Type 2 asphalt (75°C to 83°C or 167°F to 181°F) but those of Samples A and D exceeded the upper boundary.

The softening point of all samples fell within the CSA specification for Type 2 after the heating process. The heating process resulted in significant softening point fallback for Samples A and D. Originally, the softening points of these samples were higher than the specified CSA range, but they fell back into the specified range after the heating process. The other three samples (B, C and E) were less sensitive to the heating process (±1.5°C or ±3°F change in softening point). No significant difference in softening point (within 2°C or 4°F) was found between heating the samples under Open and Closed conditions.

Table 1 Softening points of the asphalt samples (the numbers in the brackets represent changes in softening point after heating).

Asphalt	Control		Open				Close			
Sample	°C	°F	°C	Δt	°F	Δt	°C	Δt	°F	Δt
A	85.0*	185*	80.0	(-5.0)	176	(-9)	78.5	(-6.5)	173	(-12)
B	75.0	167	76.0	(+1.0)	169	(+2)	74.0	(-1.0)	165	(-2)
C	76.5	170	75.0	(-1.5)	167	(-3)	75.0	(-1.5)	167	(-3)
D	86.0*	187*	81.5	(-4.5)	179	(-8)	80.5	(-5.5)	177	(-10)
E	79.0	174	80.0	(+1.0)	176	(+2)	80.5	(+1.5)	177	(+3)

* Exceeded the CSA Type 2 specification.

Viscosity

Initially the study was to include a comparison of the suppliers/manufacturers' recommended EVT with the EVT derived from testing of the control samples using ASTM D4402. However, this information was provided for only one of the five samples (see ***Appendix A***). A review of manufacturers technical literature and information provided by the various suppliers/manufacturers indicated that the recommended EVT for Type 2 asphalts at 75cP is between 188°C to 240°C (370°F to 464°F) and 173°C to 226°C (345°F to 440°F) at 125cP. All samples fell within this range before heating.

The relationship between viscosity and temperature for the asphalt samples with different heating histories are shown in Figures 4a to 4e. The best-fitted lines were obtained from regression using least squares based on the Arrhenius form equation (i.e. log[viscosity] vs1/[temperature]). Using interpolation, the temperatures at which the asphalt samples had viscosities of 125cP and 75cP were obtained and are summarized in Tables 2-3.

Table 2 Temperatures at which the asphalt samples reached a viscosity of 125cP (the numbers in the brackets represent changes in T_{125} after heating.)

	Control		Open					Close				
Samples	°C	°F	°C	^{D}t	°F	^{D}t	T_o/T_x [A]	°C	^{D}t	°F	t	T_c/T_x
A	195	383	190	(-5)	374	(-9)	.97	195	(0)	383	(0)	1.0
B	205	401	180	(-25)	356	(-45)	.87	200	(-5)	392	(-9)	.97
C	205	401	165	(-40)	329	(-72)	.80	165	(-40)	329	(-72)	.80
D	225	437	220	(-5)	428	(-9)	.97	205	(-20)	401	(-36)	.91
E	225	437	190	(-35)	374	(-63)	.84	190	(-35)	374	(-63)	.84

[A] T_o/T_x, T_c/T_x are ratios of the EVT after heating to the EVT of the control. Unity signifies no change and is an indicator of the heat sensitivity of the asphalt to heat exposure.

Table 3 Temperatures at which the asphalt samples reached a viscosity of 75cP (the numbers in the brackets represent changes in T_{75} after heating).

	Control		Open					Close				
Samples	°C	°F	°C	^{D}t	°F	^{D}t	T_o/T_x	°C	^{D}t	°F	^{D}t	T_c/T_x
A	215	419	210	(-5)	410	(-9)	.97	215	(0)	419	(0)	0
B	220	428	195	(-25)	383	(-45)	.88	210	(-10)	410	(-18)	.95
C	220	428	185	(-35)	365	(-63)	.84	195	(-25)	383	(-45)	.88
D	240	464	235	(-5)	455	(-9)	.98	220	(-20)	428	(-36)	.91
E	240	464	210	(-30)	410	(-54)	.87	210	(-30)	410	(-54)	.87

The viscosity decreased as the temperature increased as expected. However, the viscosity-temperature relationships for the five asphalt samples were different (Figure 3). At a particular temperature, the viscosity was lowest for Sample A, higher for Samples B and C, and highest for Samples D and E. This difference diminished as temperature increased. From a heating point of view, when the asphalts were heated, Sample A reached a particular viscosity at a lower temperature than Samples B and C.

Samples D and E reached that particular viscosity at a higher temperature. For example, Samples A, B and C reached the recommended viscosity for mopping (125cP) at about 200°C (392°F) and the viscosity for mechanical spreading (75cP) at around 220°C (428°F). However, Samples D and E were heated 25°C (45°F) higher to reach the same viscosities.

Figures 4a to 4e show the viscosity-temperature relationship for the asphalt samples with different heating histories. No significant difference in viscosity-temperature relationship was found between heating Sample A under Open and Closed conditions.

For Samples C and E, the viscosity became significantly less sensitive to temperature after the four hour heating. Also, the viscosity at a specific temperature decreased (T_x denotes the temperature at which an asphalt reached a viscosity of x cP). T_{125} fell by 35°C to 40°C (63°F to 72°F) after heating while T_{75} fell by 25°C to 35°C (45°F to 63°F). No difference was observed between heating under Open or Closed conditions. For Samples B and D, the viscosity became slightly less sensitive to temperature after the four hour heating. The viscosity at a specific temperature also decreased slightly. For Sample B, this decrease was more prominent for the Open condition. Both T_{125} and T_{75} fell by 25°C (45°F) for the Open samples but only by 5°C to 10°C (9°F to 18°F) for the Closed samples. However, the opposite trend was observed for Sample D, i.e. the decrease in viscosity after heating was more significant for the Closed condition. Both T_{125} and T_{75} fell by only 5°C (9°F) for the Open sample but by about 15°C (27°F) for the Closed sample.

Testing & Analysis--Phase II

In 2001 CRCA obtained four samples of Canadian Type 2 asphalt from member contractors in central and western Canada. The samples were obtained from four different supply locations. The samples were collected and prepared as those in Phase I of the study. Each asphalt sample was divided into Control, Open and Closed portions. The Open and Closed portions were heated in a convection oven at 232±10°C (450±18°F) for four hours. The effect of heating on the EVT of the asphalt samples for viscosities of 125cP and 75cP was determined and is shown in Tables 4 and 5, and Figures 5a to 5d.

Table 4 Temperatures at which the asphalt samples reached a viscosity of 125cP (the numbers in the brackets represent changes in T_{125} after heating).

	Control		Open					Close				
Samples	°C	°F	°C	^{D}t	°F	^{D}t	T_o/T_x	°C	^{D}t	°F	^{D}t	T_c/T_x
F	215	420	220	(+5)	428	(+8)	1.02	210	(-5)	410	(-8)	.97
G	230	446	215	(-15)	419	(+27)	.93	220	(+10)	428	(-18)	.95
H	230	446	235	0	446	0	1.04	230	0	446	0	1
I	210	410	210	0	410	0	1	210	0	402	0	1

The initial testing on the Control samples as received revealed a wide variability in the EVT of the asphalts with Sample I having significantly lower EVT than the other three at a viscosity of both 125cP and 75cP. This means, during heating, Sample I would reach the required viscosity for proper mopping before the rest. The results also showed that the EVTs of the samples did not change when heated at 232±10°C (450±18°F) for four hours to the same degree as when heated at 260±10°C (500±18°F) for four hours. The shape of the viscosity-temperature curve for Sample G and H did not change after heating under both Open and Closed conditions. While the EVT (both T_{125} and T_{75}) were reduced by 10 to 15°C (18 to 27°F) after heating for Sample G, the change in EVT for Sample H was insignificant (less than 5°C [9°F]) within experimental error). The viscosity of Sample F became slightly less sensitive to temperature after heating in Closed condition but this change did not affect its EVT (less than 5°C [9°F]). The viscosity-temperature curve shifted to the right (i.e. slight increase in viscosity a particular temperature) after heating in Open condition. However, this increase in EVT was insignificant. Sample I became noticeably less sensitive to temperature after heating in both Open and Closed condition.

Table 5 Temperatures at which the asphalt samples reached a viscosity of 75cP (the numbers in the brackets represent changes in T_{75} after heating).

Samples	Control		Open					Close				
	°C	°F	°C	^{D}t	°F	^{D}t	T_o/T_x	°C	^{D}t	°F	^{D}t	T_c/T_x
F	230	446	235	(+5)	454	(+8)	1.02	230	0	446	0	1
G	245	473	230	(-15)	446	(-27)	.94	235	(+10)	455	(-18)	.96
H	245	473	250	(+5)	482	(+9)	1.02	250	(+5)	482	(+9)	1.02
I	220	428	220	0	428	0	1	220	0	428	0	1

Discussion

Recommended EVT temperatures are used in the construction of BUR systems to provide a window to the installer for the application of asphalt to achieve optimal adhesion between the bitumen and reinforcing plies. They are used as quality control instruments and establish temperature application limits. During cold weather (winter) installations, it may be necessary to heat the asphalt in the kettle at temperatures significantly higher than the recommended EVT. Heating the asphalt to these temperatures may affect the physical properties of the asphalt and particularly their EVTs.

As a result of this study, the following observations were made:

1. Softening point (SP) fallback is a poor indicator of changes to the application properties of the asphalt after heating. Upon heating samples at 260±10°C (500±18°F) for four hours, all samples had softening points within the range

prescribed in the material standard (CSA A123.4-98). However, the EVT of some of the samples were significantly altered.

2. There was no clear relationship between SP fallback and change in EVT after heat conditioning. Some samples that exhibited good SP stability after heating exposure experienced a large change in EVT.
3. The viscosity of some of the asphalt samples exhibited greater sensitivity to heating than others. The viscosity stability of the samples could not be predicted based on the initial viscosity testing of the control samples.
4. There appears to be a narrow range of time and temperature (±232°C to 260°C [±450°F to 500°F]) wherein significant changes to EVT may occur. Normal heating temperatures in the field appear to fall within this range.
5. The temperatures required to obtain a viscosity of 75cP for some of the samples were above 235°C (455°F). Cold weather applications using mechanical spreaders may require heating of the asphalt at or above the upper boundary of the normal heating range resulting in a change to EVT.
6. No significant difference in terms of the rheological properties of the asphalt samples heated under Open or Closed conditions were observed except for Samples B and D. These samples were heated at 260±10°C (500±18°F) for four hours and the difference in EVT between the Open and Close conditions was about 15°C (27°F).

Recommendations

1. The EVT of particular asphalts may have to be adjusted in the field as a result of heating time and temperature, particularly during cold weather installations.
2. EVT changes can be affected by heating time and temperature and the sensitivity of the asphalt to heat exposure. Information regarding the viscosity stability of particular asphalts would be useful to the applicator in the field. Cullen suggested that this information could be provided by a viscosity ratio value calculated on the basis of viscosity values obtained before and after a heat exposure test.[13]
3. During cold weather installations, applicators should attempt to use viscosity stable asphalts.
4. Suppliers/manufacturers should test asphalts for sensitivity to heat exposure and make this information available to the purchaser.

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Appendix A

Product Information

CSA Standard A123.4-98 requires that EVT information for roofing asphalt be clearly marked on labels in the case of kegs or on the bills of lading in the case of bulk asphalt. Copies of bills of lading received with the samples revealed that only one supplier/manufacturer provided EVT information in accordance with the standard. Even in this instance, there was some confusion regarding the EVT information as the units of viscosity were expressed in centistokes as opposed to centipoises. In order to determine whether the failure to provide EVT information on bills of lading was an anomaly or standard industry practice, CRCA obtained more than 35 copies of bills of lading from contractors in central and western Canada. The shipping documents, from five different suppliers/manufacturers covered a time frame from May 1999 to May 2002. Only one supplier/manufacturer provided EVT information on the bills of lading. Information that was provided by all suppliers/manufacturers included the type of asphalt, weight of the shipment, location and time of pickup. One supplier/manufacturer also provided the asphalt temperature at pickup. It was noted that in the case where EVT information had been provided, the recommended EVT value was the same on all the bills of lading covering the entire two-year period.

The lack of EVT information on the reviewed bills of lading prompted CRCA to contact the five suppliers/manufacturers. Each was asked to provide information as to what test method it employed to measure viscosity of their roofing asphalt and how often viscosity measurement were carried out.

Supplier/manufacturer 1, who included EVT information on its bills of lading also provided a data sheet for its Canadian Type 2 roofing asphalt. The data sheet refers to ASTM D2170 as the test method employed for viscosity measurements.[1] This standard states that the method covers procedures for the determination of kinematic viscosity of liquid asphalts at 60°C (140°F). It also states that though the test method may be used at other temperatures, the precision is based on liquid asphalts at 60°C (140°F). The data sheets indicate a typical viscosity value of 350cst at 175°C (347°F) and 175cst at 200°C (392°F). No EVT information was given for viscosities of 75cP and 125cP. This supplier/manufacturer gave no indication as to how often testing was undertaken.

Supplier/manufacturer 2 provided technical data sheets for all asphalt types from their facilities. Each asphalt type from each supply location had a specific EVT. This supplier/manufacturer indicated that it used ASTM D4402 to measure EVT. They also indicated that viscosity testing was carried out on a routine basis biannually and that it intended to increase the frequency of the testing in the future.

Supplier/manufacturer 3 stated that viscosity testing using ASTM D4402 was routinely carried out in its manufacturing of asphalt roofing products but not for roofing asphalt. This supplier/manufacturer stated that the recommended EVT for roofing asphalt was based on benchmark testing that had been carried out in the past.

Supplier/manufacturer 4 provided technical data sheets from the refiner on its roofing asphalt and a series of graphs relating softening point temperatures to viscosity. The data sheets contained information regarding viscosity, softening point, flash point, penetration and durability. The supplier/manufacturer stated that the EVT were derived from this data and accompanying graphs. There was no indication that actual viscosity testing was routinely carried out.

Supplier/manufacturer 5 did not respond to the request for information.

Discussions with the suppliers/manufacturers and the information they provided indicates the following:

- Viscosity testing is not being routinely carried out by suppliers/manufacturers
- Different test methods are being used to measure viscosity
- EVT is not being clearly indicated on bills of lading
- When EVT information is provided, many of the recommended EVT values are derived from historical data, from extrapolation of data or from theoretical relationships between EVT and other physical properties

Conclusion

EVT has become a quality control instrument employed by contractors and roof inspectors to define a window of application in the construction of BUR systems. Application of asphalt within the correct EVT range is critical to the overall performance of a roof. However, the information presented suggests that, in Canada, correct EVT recommendations for particular asphalts are not being adequately determined by suppliers/manufacturers nor is such information being provided to contractors.

In those rare instances where EVT information is being supplied, the accuracy and credibility of the EVT information is questionable. In most cases, the recommended EVT for a particular asphalt has been derived from historical data that may not reflect the true properties of the asphalt being used currently. As a result, contractors are relying on general rules of thumb for EVT application guidelines.

Although the EVT concept remains technically sound, the manner in which it is currently being implemented and the apparent reluctance of many of the suppliers/manufacturers to conduct viscosity testing in a routine and consistent manner indicates that it may be of little value as a tool for quality assurance in the construction of BUR systems.

Reference

1. ASTM D2170-89, "Standard Test Method for Kinematic Viscosity of Asphalts (Bitumens)," Annual Book of ASTM Standards, Vol. 04.03,1999.

Appendix B – Graphs

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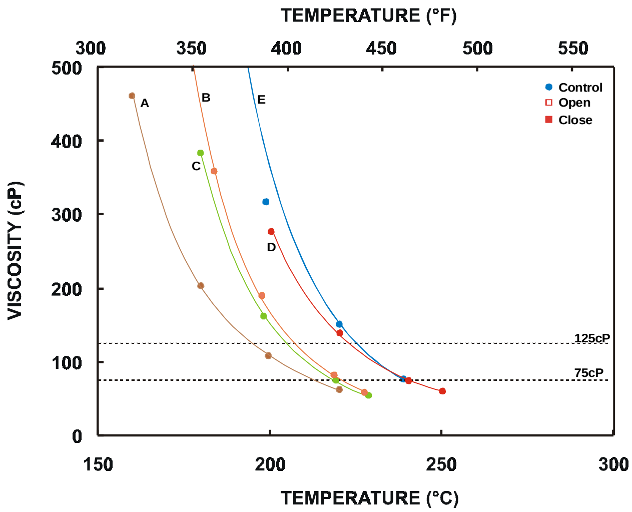

Fig. 3 Viscosity as a function of temperature for control samples.

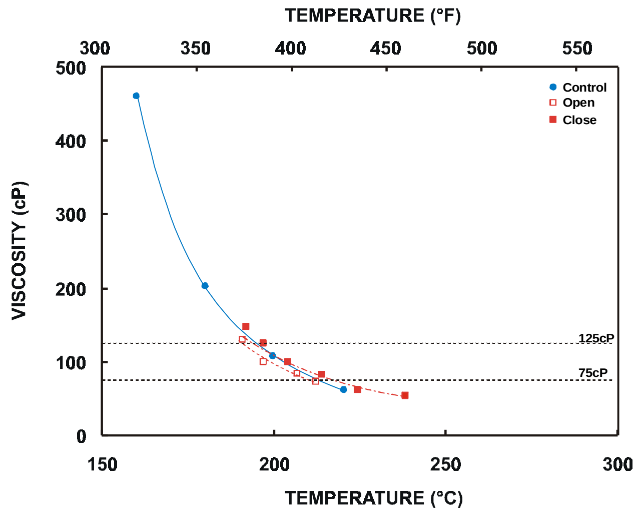

Fig. 4a Viscosity of sample A as a function of temperature before and after heating.

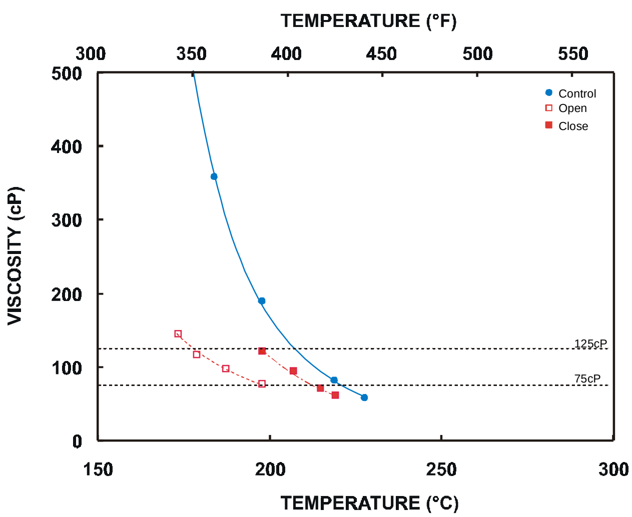

Fig. 4b Viscosity of sample B as a function of temperature before and after heating.

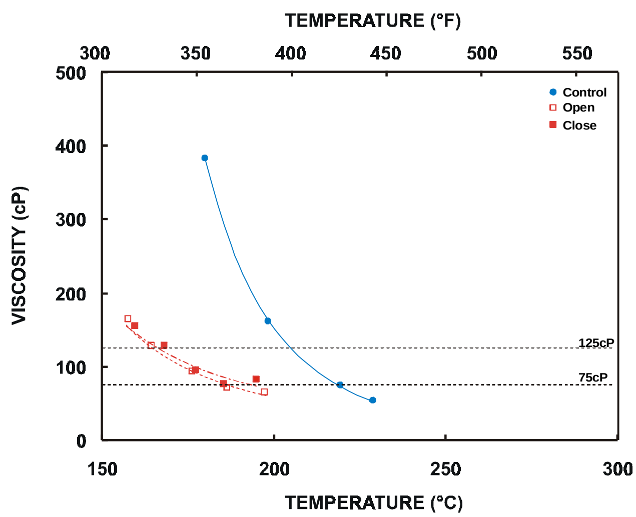

Fig. 4c Viscosity of sample C as a function of temperature before and after heating.

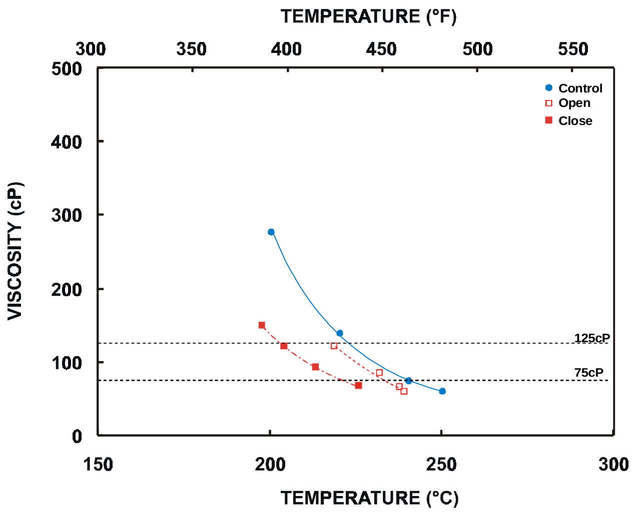

Fig. 4d Viscosity of sample D as a function of temperature before and after heating.

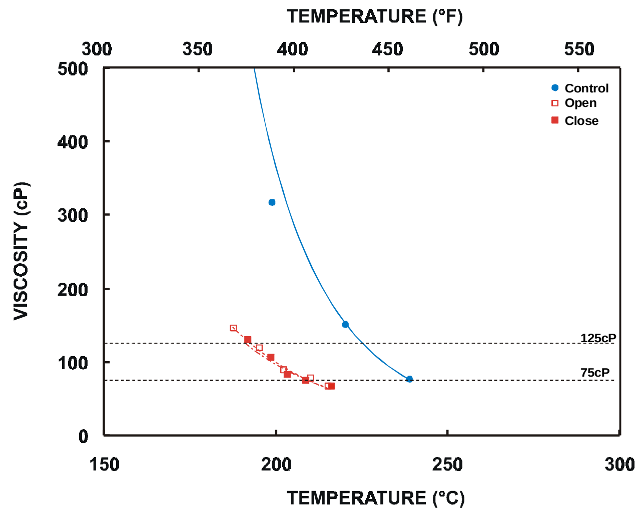

Fig. 4e Viscosity of sample E as a function of temperature before and after heating.

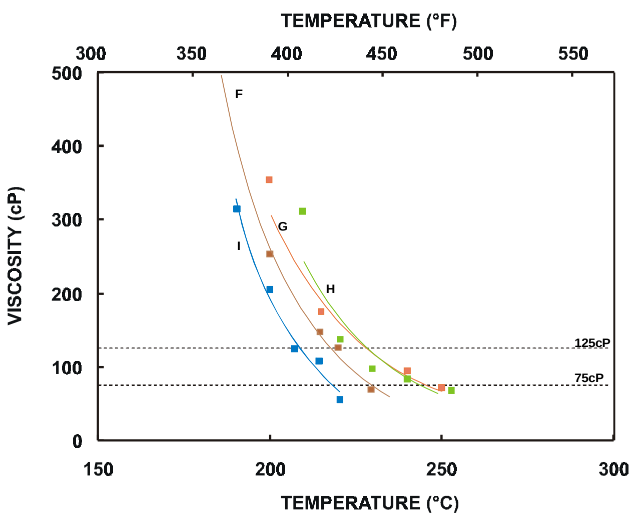

Figure 5 Phase II Control samples

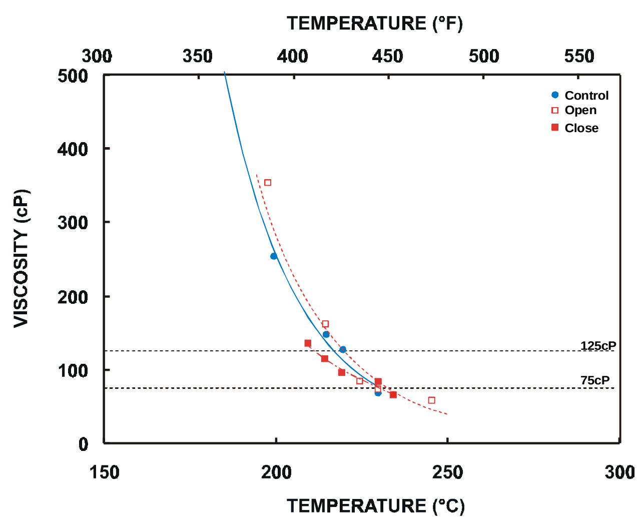

Figure 5a Sample F before and after heating.

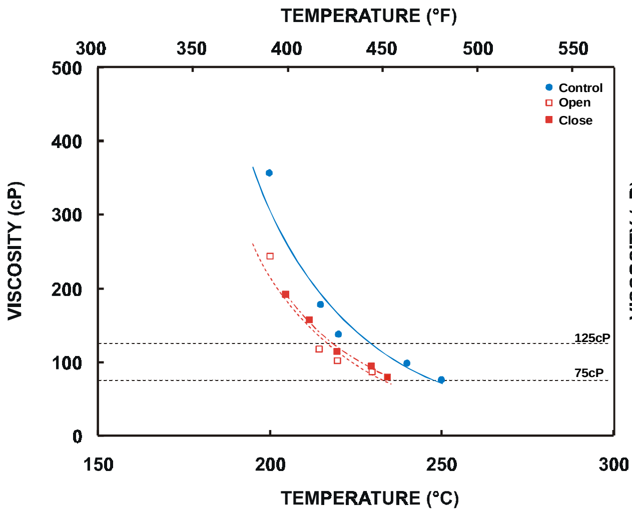

Figure 5b Sample G before and after heating.

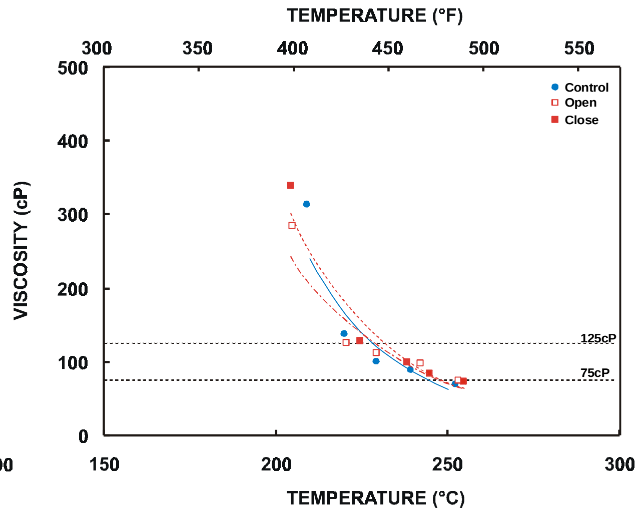

Figure 5c Sample H before and after heating

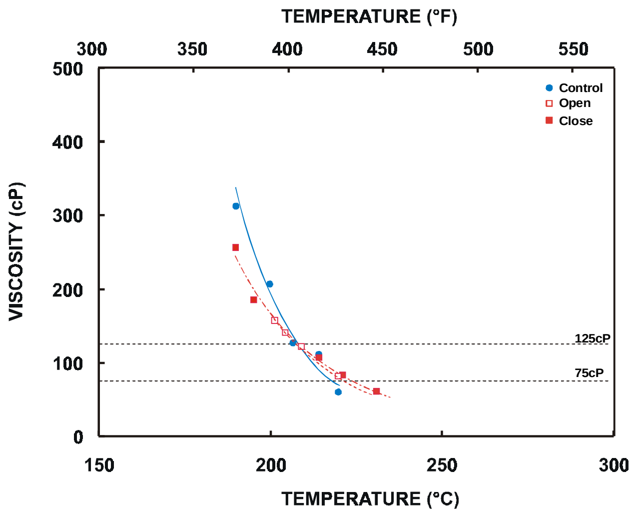

Figure 5d Sample I before and after heating